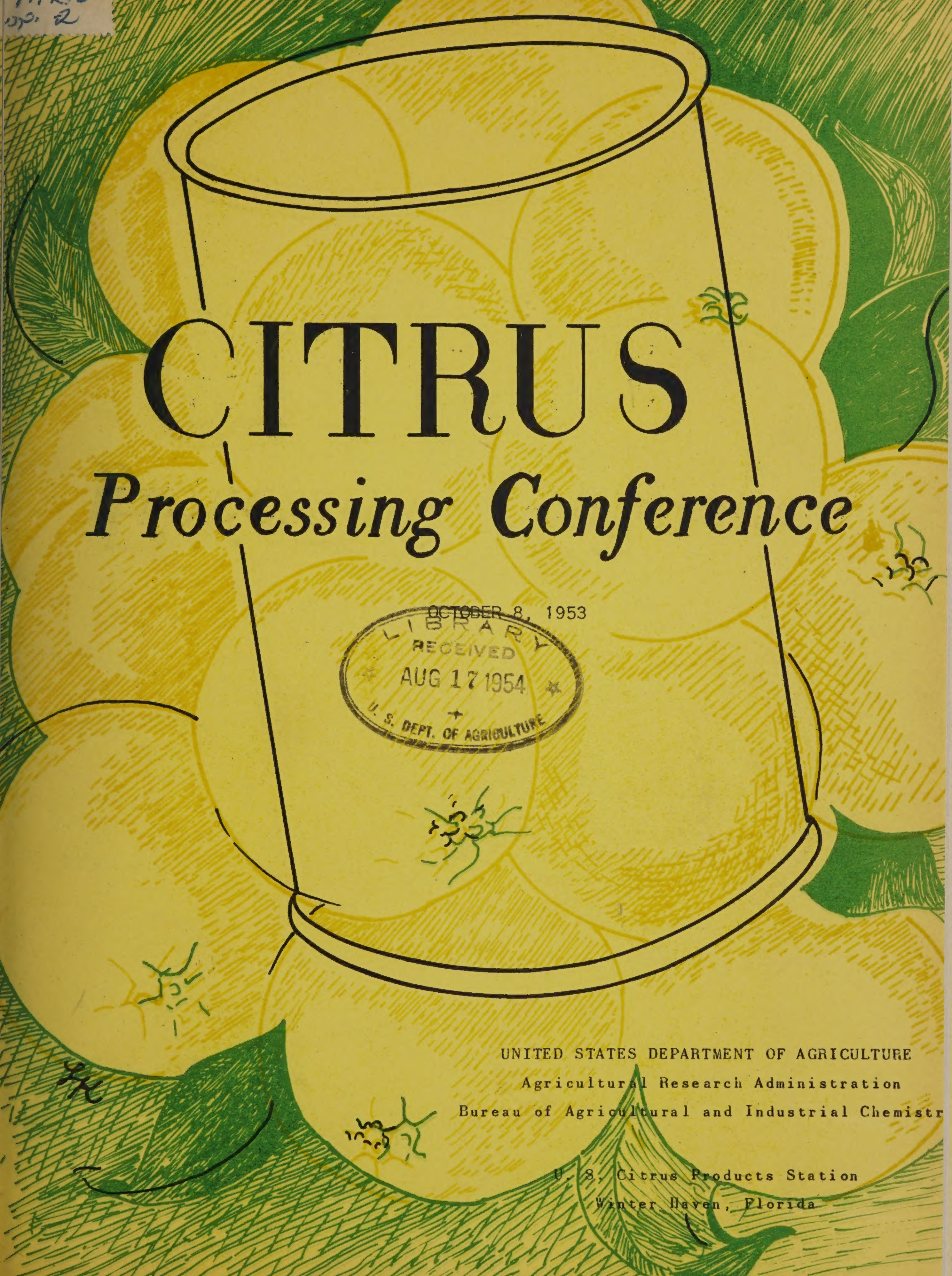

CITRUS

Processing Conference

OCTOBER 8, 1953

UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Administration
Bureau of Agricultural and Industrial Chemistr

U. S. Citrus Products Station
Winter Haven, Florida

PROGRAM AND ABSTRACTS OF PAPERS

THIRD CITRUS PROCESSING CONFERENCE

October 8, 1953

Florida Room, Citrus Building
Winter Haven, Florida

FOREWORD

This is the third Citrus Processing Conference to be called by the Bureau of Agricultural and Industrial Chemistry at its U. S. Citrus Products Station. The two previous ones, May 9, 1951, and May 27, 1952, were so successful that it seemed obvious additional ones should be held. Because people in the industry, particularly those engaged in pilot plant and other technical research usually have a little more available time in the fall, the conference data has been changed to this season of the year. It is hoped the change in date will enable and encourage greater attendance.

In the last few years, as everyone is aware, processed products have been assuming an increasing role in the utilization of the citrus crops of the nation. It is not likely, however, that the gains accruing to the citrus industry may be maintained without continued research. Therefore industry should be promptly informed of research developments in order that the results may be as promptly applied to improve processing procedures and to increase utilization. A conference provides an effective means of presenting information and of promoting the cooperation which is so essential to progress. These thoughts have motivated the organization of the 1953 conference as well as those of 1951 and 1952.

The 1953 conference, like those preceding it, has benefited from the cooperation of various organizations. The Bureau of Agricultural and Industrial Chemistry, through its field station, the U. S. Citrus Products Station, expresses appreciation to: Bureau of Plant Industry, Soils and Agricultural Engineering, U.S.D.A., Citrus Fruit Advisory Committee, U.S.D.A., Continental Can Company, Florida Canners' Association, Florida Citrus Products Research Panel, and to all others that have participated in the development of the program and contributed to its success.

The four laboratories of the Bureau of Agricultural and Industrial Chemistry which are participating in the conference are: Citrus Products Station, Winter Haven, Florida; Fruit and Vegetable Laboratory, Weslaco, Texas; Fruit and Vegetable Chemistry Laboratory, Pasadena, California; and Western Regional Research Laboratory, Albany, California.

PROGRAM
of
CITRUS PROCESSING CONFERENCE
October 8, 1953

MORNING SESSION

Chairman: M. K. Veldhuis, In Charge
U. S. Citrus Products Station
Winter Haven, Florida

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9:40	The Pasteurization and Storage of Sweetened and Unsweetened Lime Juice Owen W. Bissett, U. S. Citrus Products Station	1
10:00	Limeade Superconcentrates W. Clifford Scott, U. S. Citrus Products Station	2
10:20	Progress in Organic Chemical Investigations Lyle J. Swift, U. S. Citrus Products Station	2
10:40	The Effect of Heat-Treatment on the Cloud Stability and Flavor of Frozen Orange Concentrate Made From Pineapple and Valencia Oranges.[a] R. B. Guyer, Continental Can Company, Chicago, Ill. Owen W. Bissett, U. S. Citrus Products Station	4
11:00	Summary of Recent Progress in Citrus Research at U. S. Citrus Products Station M. K. Veldhuis, U. S. Citrus Products Station	5
11:20	Refrigerating Frozen Citrus Concentrate During Transit J. R. Winston, Bureau of Plant Industry, Soils, and Agricultural Engineering, Orlando, Florida	14

12:00 Noon Recess for lunch

a/ Report of progress of cooperative work conducted at the U. S. Citrus Products Station under a Memorandum of Understanding between the Continental Can Company, Incorporated, Chicago, Illinois, and the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture, Washington, D. C.

PROGRAM
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AFTERNOON SESSION

Chairman: Vernon H. McFarlane, Head
Fruit and Vegetable Division
Southern Regional Research Laboratory
New Orleans, Louisiana

THE PASTEURIZATION AND STORAGE OF SWEETENED AND UNSWEETENED LIME JUICE

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Citrus Products Station
Winter Haven, Florida

Canning of lime juice has been quite limited and the procedures used are those developed for orange and grapefruit juices. Lime juice is much more acid than orange or grapefruit juices and it would be expected that somewhat lower temperatures would provide adequate pasteurization. This study was conducted to obtain information on the temperature required.

Both unsweetened and sweetened lime juices were subjected to various heat-treatments ranging from 120° to 200° F., canned, and stored at 0°, 35°, and 80° F. The initial microbial population of 2000 per ml., as indicated by plate counts, is quite low in comparison with counts generally found in other citrus juices. Heat-treatment as low as 120° F. produced a marked reduction while heating to 150° F. and above destroyed most, if not all, organisms present.

Samples placed in 35° and 80° F. storage failed to develop swells. However, previous experience has shown that unheated lime juice does not always ferment.

Pectinesterase inactivation through heat-treatment was not appreciable at temperatures of 130° F. or less, but with increasing temperatures in the range of 140° to 160° F., greater percentages of the enzyme were destroyed. Following treatment at 170° F. and above, the residual pectinesterase was too small to be of significance.

Satisfactory cloud retention was observed in the unsweetened samples treated at 160° F., the heat-treatment required to destroy 95% or more of the enzyme activity. The clarifying effect of sugar in sweetened samples overshadowed any effects of the enzyme on cloud during storage.

Storage for 4-1/2 months at 80° F. induced marked changes. Unsweetened samples heated at 160° F. or less had completely clarified and a fibrous coagulum formed which was difficult to disperse. Samples that had been heated to 170° F. or above were of a milky appearance and a loose coagulum had formed which could be readily dispersed. All samples had developed a strong terpeny or "medicinal" flavor during this storage period at 80° F. and were considered unacceptable. The storage life of these products at room temperature would be very limited.

Heat-treated samples stored for 15 months at 35° F. showed no evident change in flavor. Unheated samples showed flavor changes, especially when not sweetened, but were still palatable.

These results indicate that the heating temperatures used in this experiment did not appreciably injure the flavor of the products, and that temperatures of 150° F. and above were of definite benefit.

LIMEADE SUPERCONCENTRATES

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Winter Haven, Florida

In response to the need for a more concentrated limeade than the sweetened lime juice already being manufactured commercially, the Citrus Products Station has developed two limeade concentrates: an 8-fold sweetened product requiring only water for reconstitution, and a 35-fold product requiring the addition of both sugar and water for preparation of the beverage.

The optimum characteristics of limeade as established by extensive tests are: Brix to acid ratio of 14-16:1, Brix 11°, and volatile oil 0.003-0.004%. These constants can be obtained in a concentration as high as 5-fold by sweetening single-strength juice, but concentrated juice is required to reach the maximum concentration of 8-fold. Higher concentrations of sweetened limeade concentrate are not practicable, as the solids content would have to reach 75% in order to maintain the 15:1 solids-acid ratio at 9-fold.

Since evaporation of citrus juices removes volatile flavoring oils, concentrated juices for use in beverages must be fortified with flavor-enriching materials. Tests revealed the inadequacy of steam distilled and cold-pressed lime oils, while properly prepared lime puree in sufficient quantity to provide 0.003% volatile oil in the reconstituted beverage was found to produce a beverage closely resembling that prepared from fresh juice. In order to use puree, the lime juice must be over-concentrated as in the familiar cut-back orange concentrate. For 8-fold limeade concentrate the juice need be concentrated only 2.5 fold before cutting back with puree and adding sugar, while for the 35-fold product the juice must be concentrated about 6 fold before cutting back.

Methods are given for calculating amounts of puree, concentrate and sugar required to produce limeade concentrates of desired composition.

PROGRESS IN ORGANIC CHEMICAL INVESTIGATIONS

LYLE J. SWIFT
Citrus Products Station
Winter Haven, Florida

Recent investigations conducted at the Winter Haven Station are reviewed. The main problems that have been attacked during the past 9 years are discussed briefly with particular emphasis on progress since the last Citrus Conference.

The work on lipids, most of which has been published, is summarized and correlated.

During the past two years some time has been spent in a search for a new method for the determination of pectin. While the results are still far from complete, they are quite interesting. The possibility of forming pectates with metallic ions and then estimating the quantity of pectin by measuring colorimetrically the concentration of metallic ions is being studied.

Among long-standing but still uncompleted projects is a storage experiment in which juices of different concentrations, and containing controlled amounts of limonene, terpeneless oil, and other normal fruit constituents, are compared when stored at 40° and 80° F.

During the past season, investigations were initiated on the so-called "cardboard" flavor. A quantity of volatile material having the distinctive odor and flavor has been separated and concentrated by using the essence recovery equipment. This material is being examined for its constituents.

Also during the past season, investigations have been started on constituents in the peel of citrus fruit that might contribute to off-flavor. This work is still in the preliminary stages, but the method of approach is discussed.

Publications:

The Determination of Crude Lipid in Citrus Juices. L. J. Swift. J.A.O.A.C. 29 (4), 142, (1946).

Constitution of the Juice Lipids of the Florida Valencia Orange. L. J. Swift and M. K. Veldhuis, Food Research 16 (2), 142, (1951).

Fatty Acids of the Lipids from Canned Florida Valencia Orange Juice. L. J. Swift. Food Research 17 (1), 8, (1952).

Constitution of the Lipid from Stored Valencia Orange Juice. C. W. Huskins, L. J. Swift, and M. K. Veldhuis. Food Research 17 (2), 109, (1952).

Changes in the Lipid Fraction of Valencia Orange Juice During Pasteurization. C. W. Huskins and L. J. Swift. Food Research 18 (3), 305, (1953).

Storage Changes in the Phosphorus, Nitrogen, and Fatty Acid Constituents of the Lipid in Canned Florida Valencia Orange Juice. C. W. Huskins and L. J. Swift. Food Research 18 (4), 360, (1953).

THE EFFECT OF HEAT TREATMENT ON THE CLOUD STABILITY AND FLAVOR OF FROZEN ORANGE CONCENTRATE MADE FROM PINEAPPLE AND VALENCIA ORANGES

R. B. GUYER AND W. M. MILLER
Research Division, Continental Can Company, Inc.

O. W. BISSETT AND M. K. VELDHUIS
Citrus Products Station
Winter Haven, Florida

During the past several years the use of some type of heat treatment for stabilization purposes has become quite widespread in the manufacture of frozen orange concentrate. Although a good deal of information has been reported on the fundamental aspects of heat stabilization, such as enzyme inactivation, pulp content, and pectin content, there is still need for additional studies on the commercial application of the stabilizing treatment. This need was further borne out by the results of work conducted by the Research Division, Continental Can Company, Inc., during the 1952 season in California. As a result, further investigations have been undertaken this year in cooperation with the U. S. Citrus Products Station.

The pilot plant procedure followed for the introduction of all variables included washing, extraction, heat treatment through a turbulent flow, small tube heat exchanger, cooling, and concentration of the stabilized juice to 55° Brix in a pilot plant evaporator. Unheated juice was used to cutback to 42° Brix. The variables under investigation included times of from 1 to 120 seconds at temperatures ranging from 140° F. to 210° F. Both Pineapple and Valencia varieties were investigated. The effects of all variables on pectinesterase activity, cloud stability, flavor, and microbiological population were appraised. The analytical work has only recently been completed and there has not been sufficient time to make a complete evaluation of all of the data obtained. A preliminary survey reveals the following points:

1. Concentrate made from Valencia oranges has less tendency to separate than does concentrate made from Pineapple oranges.

2. Change of the temperature of the stabilizing heat treatment appears to have a more pronounced effect on the cloud stability than does the change of time for any one temperature.

3. Temperatures of stabilization below 160° F. are not nearly as effective for cloud stability as temperatures above 160° F. This was especially true of the Pineapple variety variables.

4. Heat treatment of 190° F. to 210° F. resulted in complete cloud stability for at least 72 days when stored at 40° F.

5. No significant difference in flavor could be noted between any of the heat treated samples and the controls.

SUMMARY OF RECENT PROGRESS IN CITRUS RESEARCH AT THE U. S. CITRUS PRODUCTS STATION

M. K. VELDHUIS
Citrus Products Station
Winter Haven, Florida

Investigations being conducted at the U. S. Citrus Products Station but not discussed elsewhere during the conference are reviewed. Included are two papers which are to be presented at the coming Florida State Horticultural Society meeting in November. One is entitled "Effect of Concentration of Orange Juice and Temperature of Storage on Growth and Survival of Microorganisms". In the study reported in this paper fermentations were noted in concentrates up to 65° Brix in 50° and 60° F. storage and up to 45° Brix at 35° F. Data on the survival of coliforms and on slime and gum forming microorganisms were also obtained. The second of these papers is entitled "Notes on Factors Associated with Gelation in Frozen Concentrated Orange Juice". It deals with the results of some analyses for pectin and pectinesterase activity in commercial concentrates. Evidence of some correlation between gelation and pectinesterase and pectin values was obtained, but other factors also appear to be important.

Investigations have been conducted on the possibility of using a modification of the Boehi process to keep unheated orange juice from spoiling. Carbondioxide under 125 pounds per square inch gauge pressure was used. It was found that carbon dioxide decreases the rate of clarification in single-strength juice and 65° Brix concentrates, but does not entirely prevent it. In 40° F. storage, the rate was much slower than at 60° F., but clarification did occur at a rate too high to make the use of carbon dioxide interesting for this purpose. In addition to the clarification effects, off-flavors were noted from the growth of microorganisms. The carbon dioxide was effective in inhibiting yeast growth, but not all bacteria.

During the past year a study was initiated on growth rates of spoilage organisms as affected by pH and concentration of citrus juices. This study is still in the preliminary stages, but the technique has been worked out and some interesting values have been obtained. Some of the microbial cultures (Leuconostoc species and Lactobacillus species) being used have been isolated at the Station, and additional cultures have been obtained from other laboratories. Wide differences in the rates of growth of the different species of microorganisms have been observed.

The work with ultrasonics has been continued. The original application of high frequency sound waves, although at a comparatively low level, had caused a loss in pectinesterase in water solution, but no decrease in enzyme activity in juice or concentrate. It was considered that the results were sufficiently interesting to warrant further study of the enzyme system under increased acoustical intensity. Some time has been spent in setting up improved arrangements, but, so far, the pectinesterase in juice and concentrate has remained unaffected.

CITRUS RESEARCH AT THE U. S. FRUIT AND VEGETABLE PRODUCTS LABORATORY

F. P. GRIFFITHS
Fruit and Vegetable Products Laboratory
Weslaco, Texas

Replanting of citrus has continued in the Lower Rio Grande Valley. By the end of 1953, the Valley will have an estimated six million trees. Fully 80 percent of the new plantings will have colored fruit. Major research projects at the Weslaco laboratory are concerned with the preparation of a satisfactory canned single-strength colored juice from pink and red grapefruit, and the development of a concentrate which does not require frozen storage for preservation. Homogenization of the pulp taken from colored fruit and addition of this pulp to the extracted juice has proved the most satisfactory method of preparing a colored juice.

Work on preparation of stable concentrates indicates that double flash pasteurization, that is, flash pasteurization of the juice to partially inactivate enzymes, followed by low temperature concentration, then another flash pasteurization, and aseptic filling, is a promising method of stabilization.

Seasonal work on Mexican limes has demonstrated that a satisfactory frozen limeade base can be prepared if bitterness is avoided and low oil content maintained by care in extracting the juice. Oil may also be removed by use of deoilers.

NITROGENOUS CONSTITUENTS IN CITRUS JUICES

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Pasadena, California

Data showing that California Valencia orange juice contains nine major nitrogenous constituents including alanine, arginine, aspartic acid, asparagine, gamma-aminobutyric acid, glutamic acid, glutamine, proline, and serine, as well as three minor nitrogenous constituents, histidine, cysteine, and glutathione have been reported previously. These compounds contain essentially all of the nitrogen in filtered orange juice and compose approximately 5 to 10 percent of the total orange juice solids. The qualitative studies on the nitrogenous constituents in citrus juices have been extended by the development of a rapid, small scale paper chromatography procedure for the quantitative estimation of free amino acids in orange juice. Quantitative estimations of five amino acids (aspartic acid, glutamic acid, alanine, serine, and gamma-aminobutyric acid) in fresh and heated (12 minutes at 200° F.) canned, single strength orange juice indicated that 20% of the aspartic acid and serine and 50% of the alanine and glutamic acid, but none of the gamma-aminobutyric acid were destroyed during the heat treatment. No further changes were observed in the heated juice after storage for 2 months at 100° F.

It is of interest that the color of orange juice does not change significantly during heating, but that darkening progresses gradually during storage at room and elevated temperatures. It would appear that if a "Maillard Browning" reaction occurs in heated citrus juices, as has been suggested by other workers, the primary decomposition products of the amino acids initiate the "Browning Reaction", but are not in themselves responsible for darkening. These observations are consistent with present-day theories of browning induced by amino acid-carbohydrate interactions.

The same five amino acids were estimated in the juices obtained from California Valencia oranges harvested at various stages of maturity during each of the 1950, 1951 and 1952 seasons. The data obtained confirmed previous preliminary observations which showed wide, regular variations in the amount of amino acids, and especially gamma-aminobutyric acid, present in the juice of fruit harvested at different stages of maturity. The ratio of gamma-aminobutyric acid: acid appears to be a more sensitive index of maturity and organoleptic quality than the currently employed Brix: acid maturity index. However, a large scale comprehensive study would be necessary in order to compare the correlation of any new maturity index and the present index with organoleptic quality.

Work is being continued on the effect of processing on the amino acid content of orange and other citrus juices as well as on the quantitative variations of the nitrogenous constituents with the maturity of California Valencia oranges. Samples of fresh and pasteurized single strength and concentrated lemon juice with and without sulfur dioxide have been stored at 0° F. and 75° F. for periods up to two years. The nitrogenous constituents in the fresh and processed lemon juice samples will be estimated by quantitative paper chromatography to determine further the significance of these constituents in the darkening of lemon juice.

Publications:

Determination of Cysteine and Glutathione in Citrus Juices by Filter Paper Chromatography. J. M. Miller and L. B. Rockland. Arch. Biochem. & Biophys. 40, 416 (1952).

Nitrogenous Constituents in Citrus Fruits. I. Some Free Amino Acids in Citrus Juices Determined by Small-Scale Filter-Paper Chromatography. J. C. Underwood and L. B. Rockland. Food Research 18, 17 (1953).

Studies on Small Scale Filter Paper Chromatography. I. Factors Affecting the Separation and Sequence of Amino Acids. L. B. Rockland and J. C. Underwood. Abstracts of Papers, 123rd Meeting of the American Chemical Society, Los Angeles, California, March 1953.

Studies on Small Scale Filter Paper Chromatography. II. A Rapid Two-Dimensional Procedure. L. B. Rockland and J. C. Underwood. Abstracts of Papers, 123rd Meeting of the American Chemical Society, Los Angeles, California, March, 1953.

FLAVONOID CONSTITUENTS IN CITRUS JUICES

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The darkening of lemon products stored at room temperature is one of the principal problems of the lemon processing industry. Although the darkening of lime juice has been attributed to chemical reactions involving flavonoid compounds, no direct proof has been reported to substantiate this claim and the possible role of flavonoid compounds in the browning of lemon juice has not been previously explored.

Procedures have been developed for the extraction of flavonoid compounds from the juice and tissues of fresh lemons. These extracts have been partially fractionated by columnar chromatography into several bands, one of which darkened measurably within two hours at room temperature. It has been shown that the darkening of this fraction corresponds with a loss in flavonoid activity indicating rapid oxidation of the unknown compound. Chromatographic examination of pasteurized, concentrated lemon juice stored for 2 years at room temperature, after which it was brown-black in color, indicated that a significant portion of the unknown flavonoid had been destroyed. Work is in progress on the isolation and identification of the labile flavonoid.

ESTIMATION OF BIPHENYL IN TREATED CITRUS FRUITS

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Fruit and Vegetable Chemistry Laboratory
Pasadena, California

The practice of using biphenyl as a fungistatic agent during the shipping of citrus fruit has created a need for a rapid and precise analytical method for the determination of this compound in juice and other citrus products.

Using the chromatostrip technique of Kirchner, Miller, and Keller, a chromatographic method has been developed for the separation of biphenyl from interfering citrus oils. Two methods of measuring the amount of biphenyl have been employed. The visual method is more convenient and applicable in the field or wherever an ultraviolet spectrophotometer is not available. For the alternative procedure the biphenyl spot is eluted from the strip and measured in an ultraviolet spectrophotometer at 248 mu.

Samples are steam distilled in a modified Clevenger apparatus and the oil-biphenyl mixture is collected in 1 ml. of normal heptane or isooctane. Appropriate dilutions of the heptane or isooctane solution are spotted on the chromatostrips in .005, 0.01, 0.02, and 0.05 ml. aliquots. The chromatostrips are developed in petroleum ether or n-hexane and examined under

ultraviolet light. For the visual method the strip is selected which has the lowest concentration at which a biphenyl spot can be observed. This strip will then contain 0.55 micrograms of biphenyl. Knowing the dilution employed and the amount of solution spotted on the strip, the total amount of biphenyl in the sample may be calculated.

With the spectrophotometric method, fewer strips may be run and any strip which has a suitable biphenyl spot can be selected for the spectrophotometric determination. The biphenyl spot is scraped from the strip and then eluted with alcohol. From the spectrophotometric measurement of the eluted material the concentration of biphenyl may be interpolated from a standard curve.

As shown by Table I, the precision varies from ±0.03 mg. for samples containing less than 1 mg. of biphenyl to ±5.2 mg. for samples containing 50 to 70 mg. of biphenyl for the spectrophotometric method. The precision for the visual method is approximately half that for the instrument method.

Table I

Recovery of Added Biphenyl

Range in Total Mg. in Sample	P.P.M. in Sample	Visual No. Obs.	Visual Ave. Deviation	Spectrophotometric No. Obs.	Spectrophotometric Ave. Deviation
# 0-1	0-0.7	7	±0.08 mg.	3	±0.03 mg.
# 1-10	0.7-7.0	12	±0.43	11	±0.09
# 10-20	7-13	2	±1.9	1	±0.70
° 50-70	500-700	2	±9.4	7	±5.2

Juice samples, mg. per 1500 ml. of juice.
° Peel samples, mg. per 100 gms. of peel.

SUPERCONCENTRATED FROZEN ORANGE JUICE

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Pasadena, California

Investigations reported previously demonstrated that the volatile flavor and aroma of orange and grapefruit concentrate are due principally to peel oil. This finding was applied to the production of full-flavored high density concentrates prepared by peel oil fortification. Instead of diluting evaporated concentrate with "cut-back" juice, appropriate amounts of high quality cold-pressed peel oil were added to 4-fold, 5-fold, 6-fold, and 7-fold evaporator concentrates.

Storage studies have now been completed in which the cloud and flavor stabilities of the 5-, 6-, and 7-fold concentrates were compared with those of the 4-fold concentrate stored at temperatures of 0°, 20° and 40° F. Results of the storage experiments showed that at elevated temperatures (20° and 40° F.), increasing cloud stability was obtained with increasing concentration above 4-fold. At the 6-fold level of concentration and above the cloud stability obtained in the unheated concentrates was about the same as that obtained in 4-fold concentrate stabilized by heating the evaporator feed juice for approximately one second at 150° F. Flavor stability of high density concentrates was also found to be better than that of the usual 4-fold product after storage at 20° and 40° F.

In addition to increased cloud and flavor stability, high density concentrates possess the added advantages of reduced bulk, lower freezing point, and greater ease of reconstitution.

Publication:

Flavor fortified high density frozen citrus concentrates. R. G. Rice, G. J. Keller, R. J. McColloch, and E. A. Beavens. Abstracts of Papers, 123rd Meeting of the American Chemical Society, Los Angeles, California, March 1953.

APPLICATION OF STEAM INJECTION HEATING FOR STABILIZING CITRUS CONCENTRATES

G. J. KELLER, R. G. RICE, AND R. J. MCCOLLOCH
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Studies have been undertaken to determine the efficacy of direct steam injection heating of citrus juices to increase cloud stability in frozen concentrate manufacture. These studies have included investigations of the time-temperature effects of steam injection heating, and the inter-relationship between time and temperature of heating and the concentration of product at time of heating. Conditions studied included temperatures of 150°, 175° and 200° F. for 1- and 20- second holding times, applied to juices of 12°, 27°, 43° and 58° Brix concentration. After heat treatment all samples were adjusted by concentration or dilution to 13° Brix.

These studies indicated that the concentration of juice at time of heat treatment has a marked effect on pectinesterase inactivation and cloud stability. In general, heating at any of the higher juice concentrations produced greater pectinesterase inactivation and greater cloud stability than in single strength juice at the same temperature. It would appear

that a comparatively mild heat treatment applied to concentrated juice may achieve acceptable cloud stability without flavor damage to the product. There is some evidence to indicate that at certain "critical" temperatures, increased holding times may adversely affect cloud stability.

In general, cloud stability increased with increasing destruction of pectinesterase. However, comparisons of data on cloud stability versus residual pectinesterase activity after heat treatment failed to reveal a direct quantitative relationship between cloud stabilization and pectinesterase inactivation.

TIME-TEMPERATURE TOLERANCE STUDIES ON FROZEN ORANGE JUICE CONCENTRATES

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During the past two years five lots of commercially frozen orange juice concentrate have been subjected to storage schedules approximating the time-temperature history they may undergo under actual conditions. In these experiments lots of commercially frozen concentrate are exposed to constant and cyclic temperature patterns over a twelve-months storage period. These temperature-time variations were compiled from actual transportation and warehousing records representing the most common deviations from the desired 0° F. storage. These temperatures are cumulated and permutated throughout the experiment so that samples of concentrate are obtained representing all possible combinations of simulated transportation, warehousing and retail temperature histories.

During these experiments samples are removed at the beginning and end of each temperature fluctuation and analyzed for changes in flavor, cloud and other chemical and physical properties which may reflect loss of quality. Because of the variability of orange concentrates from year to year, it is expected that specific time-temperature limits can only be established after many samples have been evaluated over a period of several seasons.

In the samples of frozen orange juice concentrates which have been examined, flavor has been found to be relatively stable. Perceptible damage to the flavor has appeared only under the more severe of the temperature histories to which the samples have been subjected. Loss of cloud was the first serious change resulting from exposure to elevated temperature, and loss of cloud appeared much sooner than detectable flavor changes. Even in those concentrates partially heat-treated to stabilize the cloud, cloud damage still tended to occur before flavor damage. The effects of temperature histories were found to be cumulative. This important finding indicated that although a flavor or cloud effect may not be observed as a result of exposure

to any given temperature, the concentrate will have undergone latent changes. When a sufficient number of temperature anomalies cumulate in a single sample, a flavor change is finally produced.

It was noted that while concentrates were stored at the 0° F. level they became increasingly sensitive with time to loss of cloud when exposed to the higher temperatures for short intervals. Cloud loss is, therefore, not a function of temperature alone, but it is also a function of the age of the sample before thawing. A differential method of cloud determination was developed which will permit an estimation of the past storage history and future storage stability of frozen orange concentrates.

Some interesting relationships were observed between pectic changes and cloud loss. "Short term" cloud loss at elevated temperatures (40° F.) was usually accompanied by a proportionate increase in oxalate soluble pectin (demethylated pectin) at the expense of water soluble (methylated) pectin. "Long term" cloud loss occurring at lower temperatures was usually accompanied by a decrease in total pectic substances. However, there is some evidence to suggest that factors other than the pectic substances are involved in cloud stability.

Publication:

Determination of pectic substances and pectic enzymes in citrus and tomato products. AIC-337. R. J. McColloch. June 1952.

THE CAROTENOIDS IN VALENCIA ORANGE JUICE

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The development of off-flavor in canned or powdered orange juices may be caused, at least in part, by changes in the lipid fraction, which includes the carotenoid pigments. The carotenoid mixture in oranges is unusually complicated and consists largely of a complex mixture of xanthophylls, most of which also contain cyclic ether groups. Counter-current distribution in a 200-tube Craig apparatus has been used to separate the carotenoids, after saponification, into 6 fractions. These were then further fractionated by chromatography. At least 20 distinct carotenoids have been found so far, as well as a number of stereoisomers; several of these appear to be either new compounds or substances of which the structure is not known.

The carotenoids which have been found are as follows: (1) hydro-carbons - phytoene, phytofluene, α- carotene, β- carotene, zeta- carotene; (2) mono-hydroxy compounds - cryptoxanthin and a substance tentatively identified as hydroxy -α - carotene; (3) diols - zeaxanthin and lutein; (4) diols containing one cyclic ether group - antheraxanthin and mutatoxanthin;

(5) diols containing two cyclic ether groups - violaxanthin, auroxanthin and two intermediate substances for which the name luteoxanthins has been proposed; (6) polyols - six components, four of which appear to be previously undescribed; the others were tentatively identified as trollixanthin and trollichrome. The presence of acidic pigments has also been confirmed.

PRODUCTION OF STABLE DEHYDRATED ORANGE JUICE

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Studies at the Western Regional Research Laboratory have resulted in the development of a powder prepared from orange juice which possess extremely good storage characteristics even at elevated temperatures. It reconstitutes readily into a very palatable beverage upon addition of water. Thus, for the first time, a product with sufficient storage stability to meet the requirements for domestic or military use has been prepared.

The process for the new product employs concentrated orange juice similar to that used for commercially packed frozen concentrate as a starting raw material. This mixture is then vacuum dried at moderately low pressures; the more expensive high-vacuum or "freeze-drying" process is unnecessary. The evacuation is conducted in such a way that the concentrate expands or "puffs" during drying. The resulting open structure makes it possible to dry the material rapidly and at a sufficiently low temperature so that flavor changes are not significant. Powder prepared by crushing the dried material to suitable size still retains the porous open structure, which permits rapid reconstitution with water. Comminuting to the proper particle size is an important part of the process, since reconstitution becomes much more difficult when the material is reduced to a very fine powder.

The original orange flavor, largely lost during dehydration, is replaced by addition of a small amount of specially prepared natural orange oil to the powder. This orange oil is first incorporated in a small amount of edible solid carrier to prevent contact of the oil with oxygen and thus to minimize flavor changes in the oil during storage.

The orange powder has a storage life in excess of six months at 100° F. and one year at 70° F. with little change in vitamin C content and only minor losses of carotene (pro-vitamin A). Deterioration at 70° F. temperature is very slow. This excellent stability is achieved in large measure through use of a packet of desiccant in each sealed can of powder to remove substantially all remaining moisture (several percent) from the powder after it has been packaged.

REFRIGERATING FROZEN CITRUS CONCENTRATE DURING TRANSIT

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Results of tests during the past few years, with refrigerator cars and trucks using different methods of refrigeration, are reported.

CARS

Four tests were conducted in eleven cars employing three methods of refrigeration.

Dry Ice: Two of three cars maintained average commodity temperatures at or slightly below loading temperatures, -3° and +0.5° F. A third car, moving in warmer weather, showed a 6.5° rise in average temperature and maximum temperatures as high as 9° F.

Water Ice and 30% Salt: One overhead-bunker car with brine-retaining tanks had a 5.5° rise in temperature, from -2° to +3.5° F., during transit, with a maximum commodity temperature of 11.5° F. In another test in a similar car, with overhead-basket type bunkers, the average temperature rose from -1° at origin to 7.5° immediately before unloading, with a maximum of 16.5° F. The superior effectiveness of the brine-retaining tanks was indicated. One end-bunker fan car, with 1000 pounds of dry ice over the load, had a 3.5° temperature rise, from 1.0° to 4.5° F. with a maximum of 9.5° F. Another fan car, with paper over the load, had a much greater temperature rise, 9°, but the average temperature at destination was 5° F., about the same as the first overhead-bunker car and the fan car with the load under dry ice. A fan car with ice and salt only had the highest rise in temperature, from an average of -1° at origin to 10.5° at destination, with maximum temperatures as high as 18.5° F.

Mechanical Refrigeration: Mechanically refrigerated cars provided the lowest temperatures. Concentrate temperatures were maintained within 0.5° of the loading averages, -1.5° and -3.5° F., in two tests, and reduced 5° from an average of 0° in another test. The maximum temperature found in these cars was 4° F.

TRUCKS

Tests were conducted during the warmer months with approximately 30 trucks, 9 using dry ice and the rest using mechanical refrigeration.

Dry Ice: In the first two tests the rise in commodity temperatures was excessive and was apparently associated with insufficient insulation of the trailer and inadequate air circulation because of a heavy tarpaulin covering the load. In a further midsummer test, with dry ice placed on top of the load and at the bottom doorway, the average commodity temperature rose from 0° F. at loading to 5.8° at unloading 63 hours later. Maximum temperatures of 12.5° and 15.5° were, however, encountered in portions of the load. Dry ice consumption was at the rate of 56.8 pounds per hour.

Tests of a new type of system, consisting of a dry ice bunker with finned surface and thermostatically controlled air circulation, installed in an insulated trailer were made in 1951 and 1952. During a shipment to Chicago in May, 1951, the rise in average commodity temperature was 2.1° between origin and destination. In the summer of 1952, tests were made with the same equipment, differing only in having a somewhat larger dry ice bunker. In a shipment to San Antonio the commodity temperature averaged -6° F. during loading and -1.4° at unloading; when it ranged from -4.5° to +1.5°. In a test to Perth Amboy, N. J., the commodity temperature averaged -1° at origin and +3.2° at destination, with a range of -.5° to +5.5°. In a third shipment to Los Angeles, the commodity with a temperature of -4.3° during loading lost 3.0° during the 89-hour trip. In a further test in October with a shipment to Philadelphia in another trailer equipped with a somewhat similar unit, the commodity temperature averaged -4.2° at the warehouse, and -1.9° immediately before unloading, with a range from -4.6° to +2.0° and +0.8°. The two highest temperatures were found at the bottom and top doorway positions, respectively.

The dry ice consumption in the five preceding tests ranged from 20 to 26 pounds per hour, with an average of 23.3 pounds. Apparently dry ice, used in conjunction with adequate air circulation, can provide satisfactory transit temperatures, but the cost may be somewhat excessive.

Mechanical Refrigeration: With regular loading and operation of the mechanically refrigerated units in the tests made in 1950, load-temperature averages at destination ranged from -2.5° to +10° F., and average rises up to 9° were recorded. Maximum temperatures of 16° and 18° were found at certain positions in the bottom layers of the load, believed to be caused by inadequate circulation of air around the load. After installation of an improved return air duct and adoption of a modified loading plan to increase air movement around the load, the average commodity temperatures were reduced from 2.5° to -0.5° in one test and to -3° in another. In a third test shifting of the load forward under the return air duct caused the average commodity temperature to rise 7.5°, but the load average at destination was only 1° because of a low loading temperature of -6.5°.

Further tests in midsummer of 1952 compared the conventional method with the modified. With an average commodity temperature of 0° at origin in both instances, the average at destination for the conventional load was 1.4°, with a range from -7° to +11.5° and +14°, the two higher temperatures being found at the bottom of the load. With the modified loading plan the average commodity temperature immediately before unloading was +1°, with a range from -1° to +3°.

Another type of mechanical unit with return air opening on the underside of the refrigerating unit rather than at the front, installed in a trailer with a 3-inch floor rack, was tested in December 1952 in a shipment to Los Angeles. At loading the temperature of the commodity ranged from -8° to -4°, with an average of -6.6°. On unloading 118 hours later, the average was -8.5°, with a range of -15° to 0°. The greatest rise in temperature during transit, 8° to 9° was found in the bottom side zones.

Tests of a new type of system, consisting of a dry ice bunker with finned surface and thermostatically controlled air circulation, installed in an insulated trailer were made in 1951 and 1952. During a shipment to Chicago in May, 1951, the rise in average commodity temperature was 2.1° between origin and destination. In the summer of 1952, tests were made with the same equipment, differing only in having a somewhat larger dry ice bunker. In a shipment to San Antonio the commodity temperature averaged -6° F. during loading and -1.4° at unloading, when it ranged from -4.5° to +1.5°. In a test to North Asbury, N. J., the commodity temperature averaged -1° at origin and +1.2° at destination, with a range of -.5° to +5.5°. In a third shipment to Los Angeles, the commodity with a temperature of -4.5° during loading lost 4.0° during the 80-hour trip. In a further test in October with a shipment to Philadelphia in another trailer equipped with a somewhat similar unit, the commodity temperature averaged -4.4° at the beginning, and -1.0° immediately before unloading, with a range from -4.5° to +2.0° and +0.8°. The two highest temperatures were found at the bottom and top corner positions, respectively.

The dry ice consumption in the five preceding tests ranged from 50 to 68 pounds per hour, with an average of 57.2 pounds. Apparently dry ice, used in conjunction with adequate air circulation, can provide satisfactory transit temperatures, but the cost may be somewhat excessive.

Mechanical Refrigeration: With regular loading and operation of the mechanically refrigerated units in the tests made in 1950, load-temperature averages at destination ranged from -2.5° to +10° F., but average rises up to 6° were recorded. Maximum temperatures of 13° and 16° were found at certain positions in the bottom layers of the load, believed to be caused by inadequate circulation of air around the load. After installation of an improved return air duct and adoption of a modified loading plan to increase air movement around the load, the average commodity temperatures were reduced from 8.5° to -0.5° in one test and to -3° in another. In a third test addition of the duct formed under the return air duct caused the average commodity temperature to rise 7.5°, but the load average at destination was only 1° because of a low loading temperature of -6.5°.

Further tests in November of 1952 compared the conventional method with the modified. With an average commodity temperature of 0° at origin in both instances, the average at destination for the conventional load was 1.4°, with a range from -7° to +11.5° and +14°, the two higher temperatures being found at the bottom of the load. With the modified loading plan the average commodity temperature immediately before unloading was +1°, with a range from -1° to +5°.

Another type of mechanical unit with return air opening in the underside of the refrigerating unit rather than at the front, installed in a trailer with a 4-inch floor rack, was tested in December 1952 in a shipment to Los Angeles. At loading the temperature of the commodity ranged from -2° to +1°, with an average of -0.7°. On unloading 110 hours later, the average was -6.5°, with a range of -15° to 0°. The greatest rise in temperature during transit, 0° to 5° was found in the bottom side areas.

In August 1953 a second test was made to New York with the same equipment, except that the floor racks were omitted and air ducts were provided along the floor at the sidewalls. The commodity had an average temperature of $+1.2^{\circ}$ at shipping point and $+0.6^{\circ}$ at destination, with a range of -6° to $+5.8^{\circ}$. Again the highest temperatures at destination were found in the bottom part of the load.

The mechanical refrigerating units now in general use give satisfactory transit temperatures when provided with means for maintaining a continuous and even flow of air around the entire load back to the refrigerating unit. This is best accomplished when trucks are equipped with flues along the sidewalls and floor racks leading to an air duct from the floor to the cooling unit above the load.